

Contains Proprietary Information: This document contains and makes reference to information that Koch Agronomic Services, LLC (KAS) considers to be commercially valuable. The fact that this document might be provided to a government agency by KAS, or made available to the public by a government agency, should not be construed to convey any right to any third-party to cite, rely upon, or otherwise make use of this document, or the data referenced herein, for any purposes. Any use other than use by the United States Environmental Protection Agency is prohibited without the express written permissions of an authorized representative of KAS.

## **REPORT**

### **Study Title:**

Determination of Storage Stability and Corrosion Characteristics of K32

**Concord Biosciences Study Number:** 035237

**Concord Biosciences Document Number:**035237-1

### **Data Requirement:**

OPPTS 830.6317 Storage Stability (August 1996)

OPPTS 830.6320 Corrosion Characteristics (August 1996)

### **Author:**

Penny Miner

### **Study Completion Date:**

19-Oct-2017

Contains Proprietary Information: This document contains and makes reference to information that Koch Agronomic Services, LLC (KAS) considers to be commercially valuable. The fact that this document might be provided to a government agency by KAS, or made available to the public by a government agency, should not be construed to convey any right to any third-party to cite, rely upon, or otherwise make use of this document, or the data referenced herein, for any purposes. Any use other than use by the United States Environmental Protection Agency is prohibited without the express written permissions of an authorized representative of KAS.



**Storage Stability and Corrosion Characteristics of K32**  
**Document No. 035237-1**

**STATEMENT OF NO DATA CONFIDENTIALITY CLAIM**

No claim of confidentiality is made for any information contained in this study on the basis of its falling within the scope of FIFRA § 10 (d) (1) (A), (B), or (C).

Company: *Koch Agronomic Services*

Company Agent: *Eric A. Searcy*

Title: *Product Regulatory Manager*

*Eric A. Searcy*  
Signature

*10/19/17*  
Date

These data are the property of Koch Agronomic Services, LLC, and, as such, are considered confidential for all purposes other than compliance with FIFRA Section 10. Submission of these data in compliance with FIFRA does not constitute a waiver of any right to confidentiality that may exist under any other statute or in any other country.

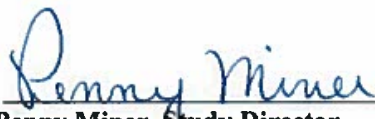
Contains Proprietary Information: This document contains and makes reference to information that Koch Agronomic Services, LLC (KAS) considers to be commercially valuable. The fact that this document might be provided to a government agency by KAS, or made available to the public by a government agency, should not be construed to convey any right to any third-party to cite, rely upon, or otherwise make use of this document, or the data referenced herein, for any purposes. Any use other than use by the United States Environmental Protection Agency is prohibited without the express written permissions of an authorized representative of KAS.



**Storage Stability and Corrosion Characteristics of K32**  
**Document No. 035237-1**

**COMPLIANCE STATEMENT**

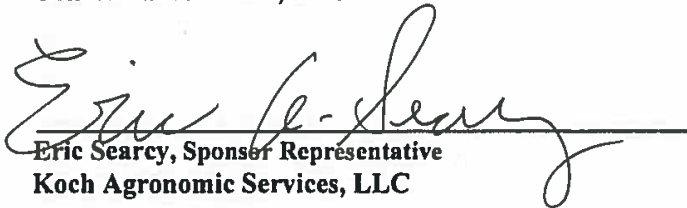
The study reported herein, "Determination of Storage Stability and Corrosion Characteristics of K32" Concord Biosciences, LLC Study Number 035237, was conducted and reported in compliance with the Good Laboratory Practice Regulations set forth in Title 40, Part 160 of the Code of Federal Regulations of the United States of America.



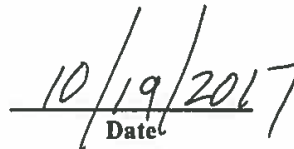
**Penny Miner, Study Director**  
**AgChem Product Development**  
**Concord Biosciences, LLC**



**Date**



**Eric Searcy, Sponsor Representative**  
**Koch Agronomic Services, LLC**



**Date**

**Submitter**

**Date**

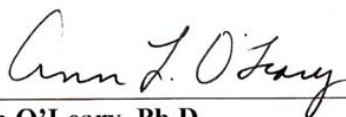


**Storage Stability and Corrosion Characteristics of K32**  
**Document No. 035237-1**

## QUALITY ASSURANCE STATEMENT

The Concord Biosciences Quality Assurance Unit has performed inspections on the study, "Determination of Storage Stability and Corrosion Characteristics of K32," Concord Biosciences Study 035237. The results of these inspections, including any findings or observations, were reported to the Study Director and Management for appropriate corrective actions on the dates listed below:

Phase Inspected	Date of Inspection	Dates Reported to the Study Director	Dates Reported to Management
Protocol	September 2, 2016	September 2, 2016	September 2, 2016
In-Study	September 22, 2017	September 22, 2017	September 22, 2017
Protocol Amendment	August 7, 2017	August 7, 2017	August 7, 2017
Data/Report	October 16 & 17, 2017	October 17, 2017	October 17, 2017

  
Ann O'Leary, Ph.D.  
Concord Biosciences Quality Assurance

October 19, 2017  
Date



**Storage Stability and Corrosion Characteristics of K32**  
**Document No. 035237-1**

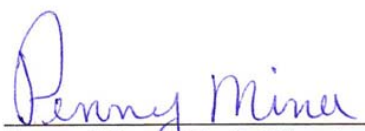
---

**APPROVALS**

Study Title: Determination of Storage Stability and Corrosion Characteristics of K32

Document Number: 035237-1

Testing Facility: Concord Biosciences, LLC  
AgChem Product Development  
10845 Wellness Way  
Concord, OH 44077



Penny Miner, Study Director  
Concord Biosciences, LLC



Date



Farhad Sayyarpour, Management  
Concord Biosciences, LLC



Date

## TABLE OF CONTENTS

	<i>Page</i>
TITLE PAGE .....	1
STATEMENT OF NO DATA CONFIDENTIALITY CLAIM.....	2
COMPLIANCE STATEMENT .....	3
QUALITY ASSURANCE STATEMENT .....	4
APPROVALS .....	5
TABLE OF CONTENTS .....	6
LIST OF FIGURES.....	7
LIST OF TABLES .....	7
LIST OF APPENDICES .....	7
CONDUCT OF THE STUDY .....	8
Sponsor .....	8
Sponsor Representative.....	8
Testing Facility .....	8
Study Director .....	8
Schedule of Events.....	8
Retention of Data .....	9
Sample Receipt and Preparation .....	9
MATERIALS AND METHODS .....	9
Test Substance .....	9
STORAGE STABILITY AND CORROSION CHARACTERISTICS PROCEDURE.....	9
HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC) .....	10
RESULTS .....	11
APPENDIX A .....	18
Temperature and Humidity Records .....	18
APPENDIX B .....	21
Protocol and Amendment .....	21

## LIST OF FIGURES

	<i>Page</i>
Figure A: Chromatogram of the K32 Formulation at Day 0 .....	12
Figure B: Chromatogram of the K32 Formulation at 3 Months, Rep 1 .....	12
Figure C: Chromatogram of the K32 Formulation at 6 Months, Rep 2 .....	13
Figure D: Chromatogram of the K32 Formulation at 9 Months, Rep 1 .....	13
Figure E: Chromatogram of the K32 Formulation at 12 Months, Rep 2 .....	14

## LIST OF TABLES

	<i>Page</i>
Table 1: Results of the Corrosion Rate (cm/hr) .....	15
Table 2: Results from the Analysis of the K32 at Day 0 (Ambient) .....	15
Table 3: Results from the Analysis of the K32 at 3 months (20° C).....	16
Table 4: Results from the Analysis of the K32 at 6 months (20° C).....	16
Table 5: Results from the Analysis of the K32 at 9 months (20° C).....	16
Table 6: Results from the Analysis of the K32 at 12 months (20° C).....	17

## LIST OF APPENDICES

	<i>Page</i>
APPENDIX A .....	18
Temperature and Humidity Records .....	18
APPENDIX B .....	21
Protocol and Amendment .....	21

Contains Proprietary Information: This document contains and makes reference to information that Koch Agronomic Services, LLC (KAS) considers to be commercially valuable. The fact that this document might be provided to a government agency by KAS, or made available to the public by a government agency, should not be construed to convey any right to any third-party to cite, rely upon, or otherwise make use of this document, or the data referenced herein, for any purposes. Any use other than use by the United States Environmental Protection Agency is prohibited without the express written permissions of an authorized representative of KAS.

## **CONDUCT OF THE STUDY**

The purpose of this study was to determine the storage stability and corrosion characteristics of K32. This study was conducted to meet the data requirements of guideline OPPTS 830.6317 (Storage Stability) and OPPTS 830.6320 (Corrosion Characteristics).

### ***SPONSOR***

Koch Agronomic Services, LLC  
2883 Miller Road  
Decatur GA 30035

### ***SPONSOR REPRESENTATIVE***

Eric Searcy  
Product Regulatory Manager  
Koch Agronomic Services, LLC  
2883 Miller Road  
Decatur GA 30035  
Phone: 770-593-6813  
Email: eric.searcy@kochind.com

### ***TESTING FACILITY***

Concord Biosciences, LLC  
AgChem Product Development  
10845 Wellness Way  
Concord, OH 44077

### ***STUDY DIRECTOR***

Penny Miner  
AgChem Product Development  
Concord Biosciences, LLC  
10845 Wellness Way  
Concord, OH 44077  
Phone: (440) 357-3718  
Fax: (440) 357-3654  
Email: penny.miner@concordbio.com

### ***SCHEDULE OF EVENTS***

Study Initiation Date:	September 12, 2016
Experimental Start Date:	September 22, 2016
Experimental Termination Date:	September 25, 2017



### ***RETENTION OF DATA***

All original data (including electronic data), or authenticated copies thereof, and a copy of the final report will be retained using appropriate storage media in the Concord Biosciences' archives upon completion of the study. The Sponsor will be contacted later to determine whether any of the data should be returned, retained or destroyed on their behalf.

### ***SAMPLE RECEIPT AND PREPARATION***

The K32 (lot 55700-30-13) was manufactured at Concord Biosciences on July 20, 2016.

The following are the significant days in this study:

9/22/2016 (Day 0)

1/16/2017 (3 months)\*

4/14/17 (6 months)\*

6/22/17 (9 months)

9/22/17 (12 months)

\*Incubation of these samples began on 10/14/2016

Temperature and humidity were monitored continuously over the 12 month interval at 20 °C.

## **MATERIALS AND METHODS**

The test substance is the test system. The study determined the stability at 20 °C over 12 months for K32.

### ***TEST SUBSTANCE***

Test Substance Name:	K32
Composition:	Reaction products of NBPT with urea and formaldehyde
Batch/Lot Number:	55700-30-13
Analyzed Concentration:	Reaction product mixtures 80.3 wt%, NBPT 17.3 wt%, water 2.4 wt%
Manufactured by:	Concord Biosciences
Date of manufacture:	July 20, 2016
Appearance:	Off-white to pale yellow gel

## **STORAGE STABILITY AND CORROSION CHARACTERISTICS PROCEDURE**

Aluminum, zinc, copper, stainless steel, mild steel, and brass coupons (1/2" x 3" x 1/16") were washed with detergent, water, alcohol, and dried in an oven at 50 °C for two hours and subsequently cleaned with absorbent paper (tissue paper).

After drying, aluminum, zinc, copper, stainless steel, mild steel and brass coupons were weighed and placed in separate six small containers in duplicate of the same composition as the commercial container (HDPE bottle) with test item (~50 g) and one commercial container in

duplicate with test item (~50 g mL) was used as control. These seven containers were considered one set. Five more sets were prepared and stored at  $20 \pm 2$  °C. The containers were weighed initially at the time of storage and then reweighed at each of the test intervals, prior to and after sampling. The samples were drawn at various intervals (0, 3, 6, 9, and 12 months).

Physical evaluation was done at each time interval (0, 3, 6, 9, and 12 months), the test substance was examined for physical changes in appearance, e.g., color and clarity. The container itself was also examined for any evidence of physical degradation, e.g., cracking, pitting, or brittleness.

One set was removed from the environmental chamber at each interval. The aluminum, zinc, copper, stainless steel, mild steel, and brass coupons were washed with detergent, water, alcohol, and dried in an oven and subsequently cleaned with absorbent paper (tissue paper). After drying of the aluminum, zinc, copper, stainless steel, mild steel, and brass coupons, visual changes on the metals were recorded. The metals were weighed and recorded for the determination of corrosion rate.

Stability was determined by comparison of the control samples (time = 0) to the samples stored in HDPE bottles at  $20 \pm 2$  °C. When analyzed, approximately 0.2 g of K32 samples was weighed into 4 mL vial and dissolved in 1 mL of acetonitrile:water, 30:70, v:v and shaken to ensure complete dissolution. Stability was determined by comparative analysis of HPLC profile of the test substance stored at  $20 \pm 2$  °C to the control sample (time = 0).

## HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

The following LC-UV method was used:

Perkin Elmer™ Series 200 Pump quaternary gradient liquid chromatograph  
Perkin Elmer™ Series 200 UV/VIS Detector  
Perkin Elmer™ Series 200 LC Autosampler  
Radiomatic FLO-ONE®\Beta Model 150TR radioactive flow detector  
(all from Perkin Elmer Instruments, Norwalk, CT)

Column: Restek Ultra C18, 3 µm, 150 mm x 4.6 mm  
Mobile Phase A: Water  
Mobile Phase B: Acetonitrile  
Flow Rate: 1.0 mL/minute  
Wavelength: 214 nm

Gradient Table 1:

Time (minutes)	Flow Rate	%A	%B
Initial	1.0	87	13
8	1.0	87	13
30	1.0	30	70
31	1.0	87	13
43	1.0	87	13

Total Time: 43 minutes  
Injection Volume: 15 µL

Contains Proprietary Information: This document contains and makes reference to information that Koch Agronomic Services, LLC (KAS) considers to be commercially valuable. The fact that this document might be provided to a government agency by KAS, or made available to the public by a government agency, should not be construed to convey any right to any third-party to cite, rely upon, or otherwise make use of this document, or the data referenced herein, for any purposes. Any use other than use by the United States Environmental Protection Agency is prohibited without the express written permissions of an authorized representative of KAS.

Data collection by Perkin Elmer Totalchrom™ (a validated system).

## RESULTS

All of the seven components of the K32 formulation were stable over the 12 months of storage at approximately 20 °C. Example chromatograms are available in [Figure A](#), [Figure B](#), [Figure C](#), [Figure D](#) and [Figure E](#). Tables of these results are contained in [Table 2](#) through [Table 6](#). The average of the replicates over the study is shown below.

Peak Area % by HPLC					
	0	3 months	6 months	9 months	12 months
#1, Component 1 (C1)	2.17	2.16	1.90	1.55	2.06
#2, Component 2 (C2)	5.46	4.79	4.09	3.64	3.67
#3, Component 3 (C3)	2.60	2.65	2.26	3.30	2.69
#4, Component 4 (C4)	21.97	22.98	23.89	26.62	25.90
#5, Component 5 (C5)	51.57	55.65	57.07	57.10	59.36
#6, Component 6 (C6)	8.14	5.84	5.37	3.87	3.22
#7, Component 7 (C7)	8.12	5.94	5.45	3.93	3.12

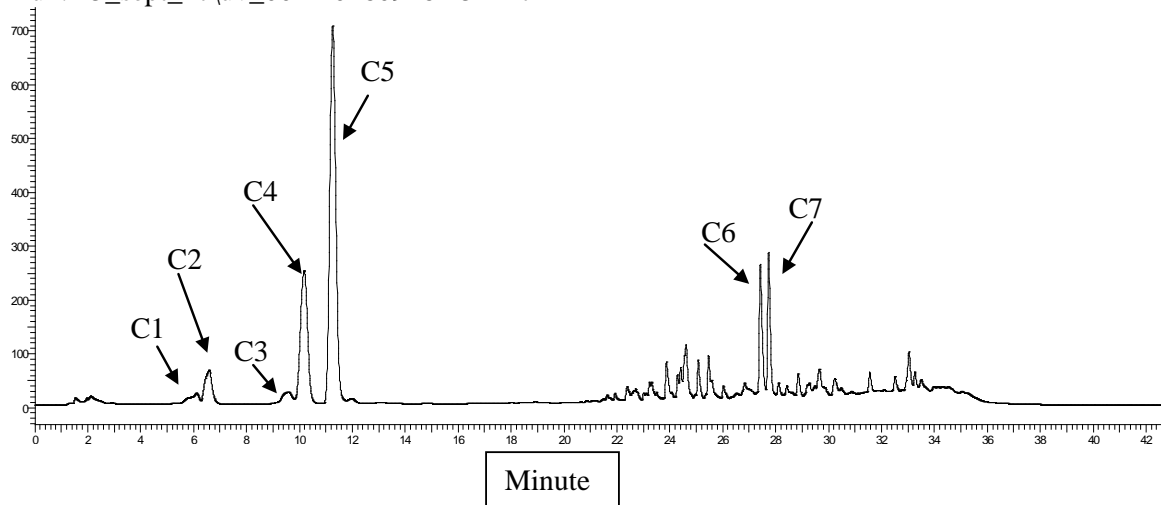
No physical changes (e.g., cracking, pitting, or brittleness) in the HDPE container itself was observed at each time point. No physical changes were observed for the K32 controls.

After the cleaning and drying of the aluminum, zinc, copper, stainless steel, mild steel, and brass coupons, visual changes on the metals were recorded in addition the test substance was examined for physical changes in appearance. The metals were also weighed and recorded for the determination of corrosion rate. These calculations of the results are contained in [Table 1](#). No changes in color were observed for the K32 that was in contact with the metal coupons. From the K32 that was in contact with the brass and copper coupons, the coupons were observed to tarnish over the 12 months. The mild steel coupons showed small rust spots at the 9- and 12-month samplings.

	Metal Changes T=3	Metal Changes T=6	Metal Changes T=9	Metal Changes T=12
Aluminum Rep-1	None	None	None	None
Aluminum Rep-2	None	None	None	None
Brass Rep-1	Tarnished	Tarnished	Tarnished	Tarnished
Brass Rep-2	Tarnished	Tarnished	Tarnished	Tarnished
Copper Rep-1	Tarnished	Tarnished	Tarnished	Tarnished
Copper Rep-2	Tarnished	Tarnished	Tarnished	Tarnished
Stainless Steel Rep-1	None	None	None	None
Stainless Steel Rep-2	None	None	None	None
Mild Steel Rep-1	None	None	small rust spots	small rust spots
Mild Steel Rep-2	None	None	small rust spots	small rust spots
Zinc Rep-1	None	None	None	None
Zinc Rep-2	None	None	None	None

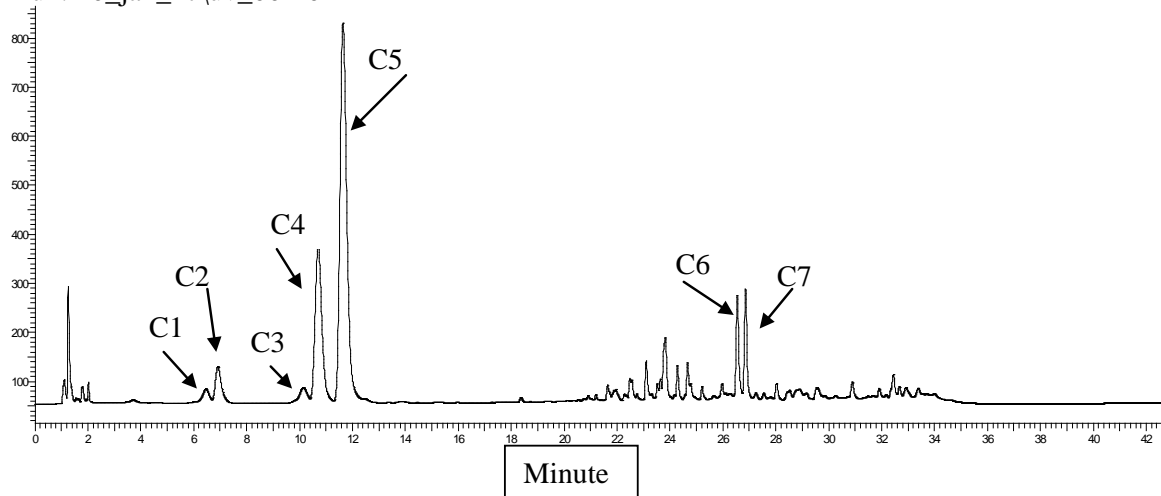
**Figure A: Chromatogram of the K32 Formulation at Day 0**

Run: 15\_sept\_17\uv\_004-20160916-151147



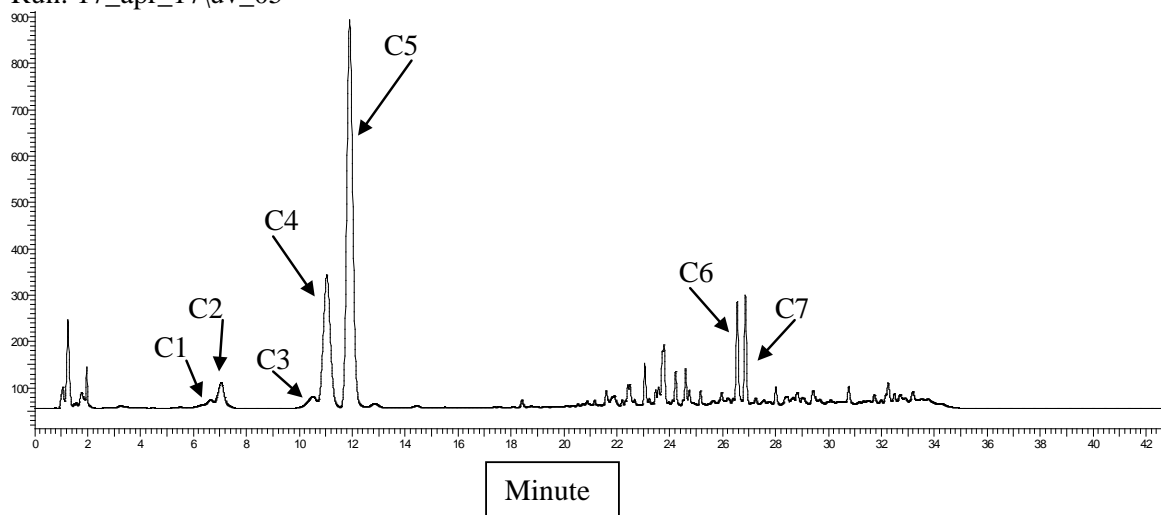
**Figure B: Chromatogram of the K32 Formulation at 3 Months, Rep 1**

Run: 20\_jan\_17\uv\_001-04



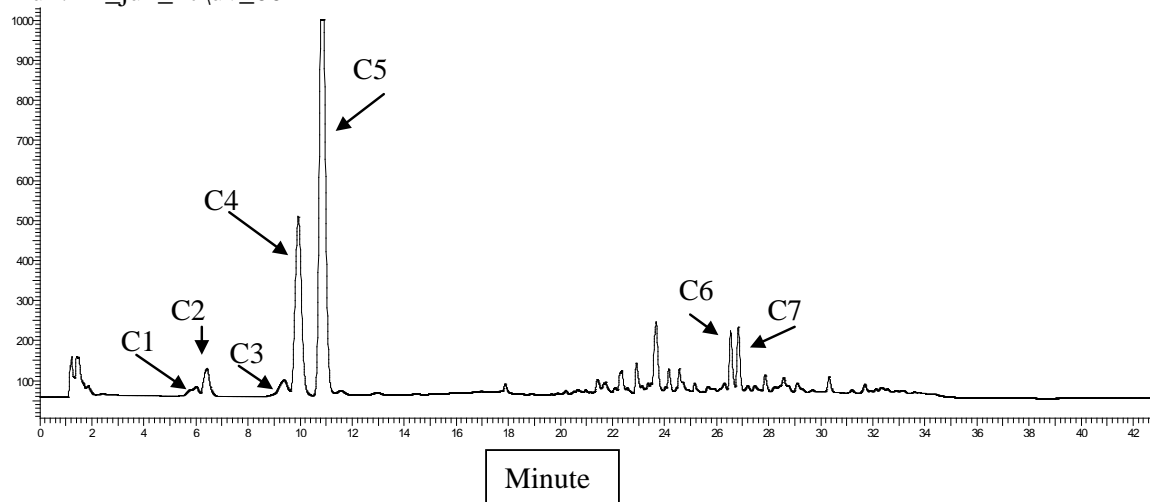
**Figure C: Chromatogram of the K32 Formulation at 6 Months, Rep 2**

Run: 17\_apr\_17\uv\_05



**Figure D: Chromatogram of the K32 Formulation at 9 Months, Rep 1**

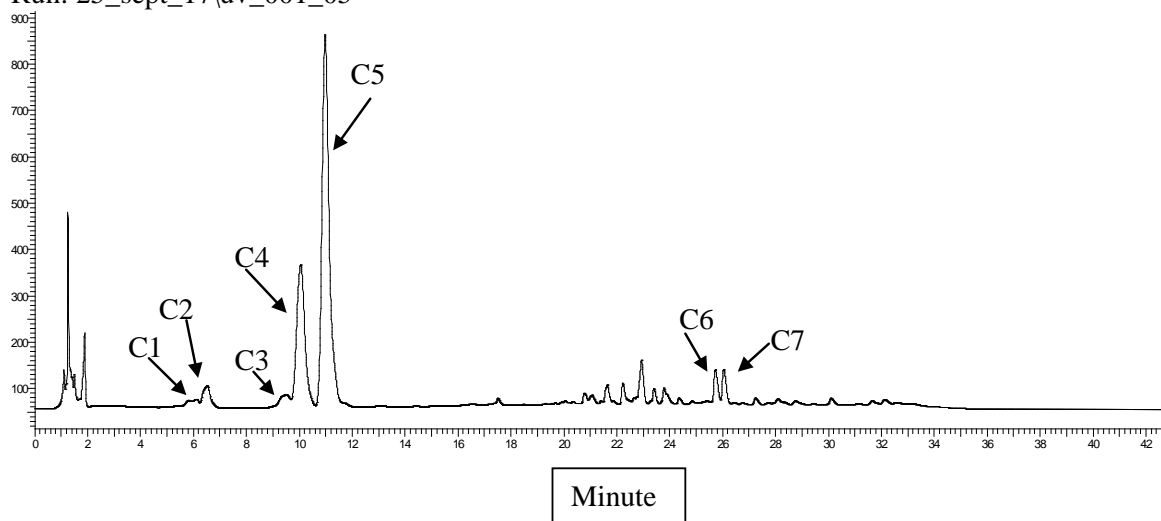
Run: 22\_jun\_17\uv\_004



Contains Proprietary Information: This document contains and makes reference to information that Koch Agronomic Services, LLC (KAS) considers to be commercially valuable. The fact that this document might be provided to a government agency by KAS, or made available to the public by a government agency, should not be construed to convey any right to any third-party to cite, rely upon, or otherwise make use of this document, or the data referenced herein, for any purposes. Any use other than use by the United States Environmental Protection Agency is prohibited without the express written permissions of an authorized representative of KAS.

**Figure E: Chromatogram of the K32 Formulation at 12 Months, Rep 2**

Run: 25\_sept\_17\uv\_001\_05



Contains Proprietary Information: This document contains and makes reference to information that Koch Agronomic Services, LLC (KAS) considers to be commercially valuable. The fact that this document might be provided to a government agency by KAS, or made available to the public by a government agency, should not be construed to convey any right to any third-party to cite, rely upon, or otherwise make use of this document, or the data referenced herein, for any purposes.

Any use other than use by the United States Environmental Protection Agency is prohibited without the express written permissions of an authorized representative of KAS.

**Table 1: Results of the Corrosion Rate (cm/hr)**

Metal	Time				
	0	3 months	6 months	9 months	12 months
Aluminum Rep-1	NA	0.03	0.09	0.02	0.01
Aluminum Rep-2	NA	0.04	NA	NA	0.02
Brass Rep-1	NA	0.02	0.09	0.01	0.02
Brass Rep-2	NA	0.10	0.02	0.01	0.03
Copper Rep-1	NA	0.05	0.09	0.05	0.02
Copper Rep-2	NA	0.06	0.03	0.05	0.04
Stainless Steel Rep-1	NA	0.05	0.02	NA	0.01
Stainless Steel Rep-2	NA	0.01	0.01	0.01	0.01
Mild Steel Rep-1	NA	NA	0.02	NA	NA
Mild Steel Rep-2	NA	0.03	0.01	0.01	NA
Zinc Rep-1	NA	0.15	0.03	NA	NA
Zinc Rep-2	NA	0.03	0.02	NA	NA

NA= not applicable and/ or calculated value  $\leq 0$ .

**Table 2: Results from the Analysis of the K32 at Day 0 (Ambient)**

	Peak Area % by HPLC*		
	Rep 1	Rep 2	Average
Peak 1	2.14	2.19	2.17
Peak 2	5.48	5.43	5.46
Peak 3	2.62	2.57	2.60
Peak 4	21.86	22.08	21.97
Peak 5	51.66	51.47	51.57
Peak 6	8.11	8.16	8.14
Peak 7	8.13	8.11	8.12

*\*Based on Total peak area*

Contains Proprietary Information: This document contains and makes reference to information that Koch Agronomic Services, LLC (KAS) considers to be commercially valuable. The fact that this document might be provided to a government agency by KAS, or made available to the public by a government agency, should not be construed to convey any right to any third-party to cite, rely upon, or otherwise make use of this document, or the data referenced herein, for any purposes. Any use other than use by the United States Environmental Protection Agency is prohibited without the express written permissions of an authorized representative of KAS.

**Table 3: Results from the Analysis of the K32 at 3 months (20° C)**

Peak Area % by HPLC*			
	Rep 1	Rep 2	Average
Peak 1	2.11	2.20	2.16
Peak 2	4.68	4.90	4.79
Peak 3	2.62	2.68	2.65
Peak 4	22.92	23.04	22.98
Peak 5	56.27	55.02	55.65
Peak 6	5.64	6.04	5.84
Peak 7	5.77	6.11	5.94

*\*Based on Total peak area*

**Table 4: Results from the Analysis of the K32 at 6 months (20° C)**

Peak Area % by HPLC*			
	Rep 1	Rep 2	Average
Peak 1	1.94	1.85	1.90
Peak 2	3.84	4.33	4.09
Peak 3	2.17	2.34	2.26
Peak 4	24.48	23.29	23.89
Peak 5	57.23	56.90	57.07
Peak 6	5.14	5.60	5.37
Peak 7	5.2	5.69	5.45

*\*Based on Total peak area*

**Table 5: Results from the Analysis of the K32 at 9 months (20° C)**

Peak Area % by HPLC*			
	Rep 1	Rep 2	Average
Peak 1	1.68	1.42	1.55
Peak 2	3.65	3.63	3.64
Peak 3	3.33	3.26	3.30
Peak 4	26.49	26.75	26.62
Peak 5	57.10	57.10	57.10
Peak 6	3.85	3.88	3.87
Peak 7	3.89	3.97	3.93

*\*Based on Total peak area*



Contains Proprietary Information: This document contains and makes reference to information that Koch Agronomic Services, LLC (KAS) considers to be commercially valuable. The fact that this document might be provided to a government agency by KAS, or made available to the public by a government agency, should not be construed to convey any right to any third-party to cite, rely upon, or otherwise make use of this document, or the data referenced herein, for any purposes.

Any use other than use by the United States Environmental Protection Agency is prohibited without the express written permissions of an authorized representative of KAS.

**Table 6: Results from the Analysis of the K32 at 12 months (20° C)**

Peak Area % by HPLC*			
	Rep 1	Rep 2	Average
Peak 1	2.05	2.07	2.06
Peak 2	3.67	3.67	3.67
Peak 3	2.66	2.71	2.69
Peak 4	25.72	26.07	25.90
Peak 5	59.17	59.54	59.36
Peak 6	3.42	3.02	3.22
Peak 7	3.30	2.93	3.12

*\*Based on Total peak area*

Contains Proprietary Information: This document contains and makes reference to information that Koch Agronomic Services, LLC (KAS) considers to be commercially valuable. The fact that this document might be provided to a government agency by KAS, or made available to the public by a government agency, should not be construed to convey any right to any third-party to cite, rely upon, or otherwise make use of this document, or the data referenced herein, for any purposes. Any use other than use by the United States Environmental Protection Agency is prohibited without the express written permissions of an authorized representative of KAS.

## **APPENDIX A**

### **Temperature and Humidity Records**

Contains Proprietary Information: This document contains and makes reference to information that Koch Agronomic Services, LLC (KAS) considers to be commercially valuable. The fact that this document might be provided to a government agency by KAS, or made available to the public by a government agency, should not be construed to convey any right to any third-party to cite, rely upon, or otherwise make use of this document, or the data referenced herein, for any purposes.

Any use other than use by the United States Environmental Protection Agency is prohibited without the express written permissions of an authorized representative of KAS.

Page 1 of 1

## Ricerca Environmental Monitoring System (REMS)

Report Type: History Chart

Sensor CMMS Number: HQ-SENB-00298

Department Equipment Name: ABS Environmental Chamber 1

Equipment CMMS Number: HQ-EC-00001

Equipment Type: Environmental Chamber

Building: A

Equipment/Space Location: A-57

Environmental Condition: Temperature

Validated Data: Sensor calibration records on file

Account Code: 02-25

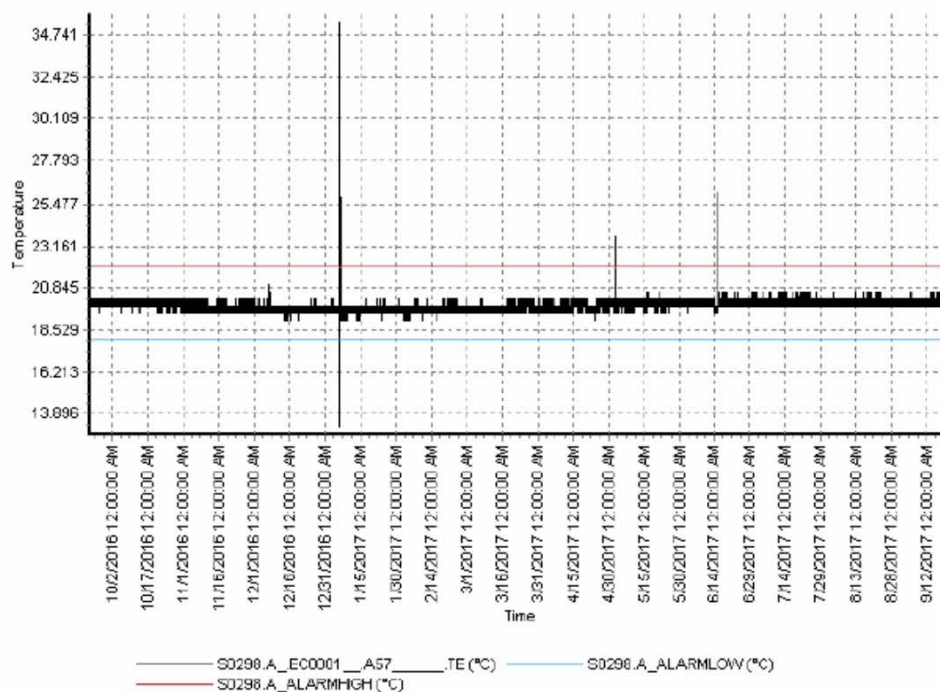
Contact Person: Walsh, Kevin

Report Date: 10/17/2017

StartDate: 9/22/2016 1:29:36 PM

EndDate: 9/22/2017 1:30:36 PM

PortionOfDay: All-Day FillDown: No



Contains Proprietary Information: This document contains and makes reference to information that Koch Agronomic Services, LLC (KAS) considers to be commercially valuable. The fact that this document might be provided to a government agency by KAS, or made available to the public by a government agency, should not be construed to convey any right to any third-party to cite, rely upon, or otherwise make use of this document, or the data referenced herein, for any purposes.

Any use other than use by the United States Environmental Protection Agency is prohibited without the express written permissions of an authorized representative of KAS.

Page 1 of 1



## Ricerca Environmental Monitoring System (REMS)

Report Type: History Chart

Sensor CMMS Number: HQ-SENB-00299

Department Equipment Name: ABS Environmental Chamber 1

Equipment CMMS Number: HQ-EC-00001

Equipment Type: Environmental Chamber

Building: A

Equipment/Space Location: A-57

Environmental Condition: Humidity

Validated Data: Sensor calibration records on file

Account Code: 02-25

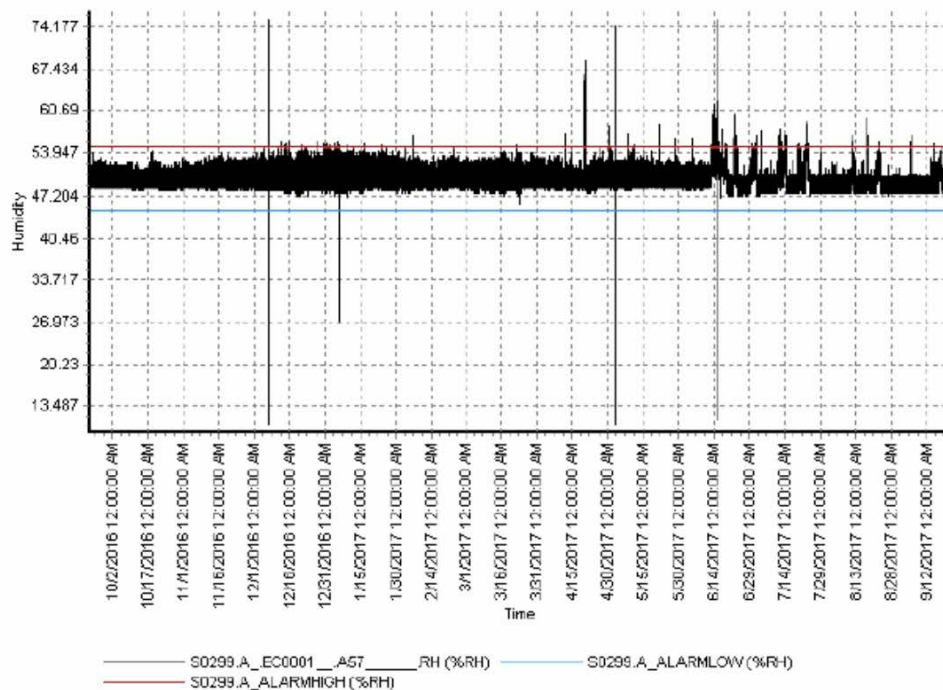
Contact Person: Walsh, Kevin

Report Date: 10/17/2017

StartDate: 9/22/2016 2:44:17 PM

EndDate: 9/22/2017 2:45:17 PM

PortionOfDay: All-Day FillDown: No



Contains Proprietary Information: This document contains and makes reference to information that Koch Agronomic Services, LLC (KAS) considers to be commercially valuable. The fact that this document might be provided to a government agency by KAS, or made available to the public by a government agency, should not be construed to convey any right to any third-party to cite, rely upon, or otherwise make use of this document, or the data referenced herein, for any purposes.

Any use other than use by the United States Environmental Protection Agency is prohibited without the express written permissions of an authorized representative of KAS.

## **APPENDIX B**

### **Protocol and Amendment**

Contains Proprietary Information: This document contains and makes reference to information that Koch Agronomic Services, LLC (KAS) considers to be commercially valuable. The fact that this document might be provided to a government agency by KAS, or made available to the public by a government agency, should not be construed to convey any right to any third-party to cite, rely upon, or otherwise make use of this document, or the data referenced herein, for any purposes. Any use other than use by the United States Environmental Protection Agency is prohibited without the express written permissions of an authorized representative of KAS.



## **PROTOCOL**

### **Study Title:**

Determination of Storage Stability and Corrosion Characteristics of K32

**Document Number:** 035237-0

### **Data Requirements:**

OPPTS 830.6317 Storage Stability (August 1996)  
OPPTS 830.6320 Corrosion Characteristics (August 1996)

### **Testing Facility:**

AgChem Product Development  
Ricerca Biosciences, LLC  
7528 Auburn Road  
Concord OH 44077

### **Study Sponsor:**

Koch Agronomic Services, LLC  
2883 Miller Road  
Decatur GA 30035



## TABLE OF CONTENTS

	<i>Page</i>
PROTOCOL .....	1
TABLE OF CONTENTS .....	2
INTRODUCTION .....	3
PURPOSE .....	3
EXPERIMENTAL INFORMATION .....	3
Schedule of Events .....	3
Sponsor .....	3
Sponsor Representative .....	3
Testing Facility .....	3
Study Director .....	3
MATERIALS AND METHODS .....	4
Storage, Distribution, and Retention .....	4
Test Substance .....	4
Characterization of the Test Substance .....	4
JUSTIFICATION FOR SELECTION OF TEST SYSTEM .....	4
EXPERIMENTAL DESIGN .....	4
Overview .....	4
Stability at an Elevated Temperature .....	5
Equipment .....	5
Reagents .....	5
Stability Storage of Sample at a Temperature of $20 \pm 2$ °C Procedure .....	5
Preparation of Samples .....	5
Physical Evaluation of Test Substance and Containers .....	5
High Performance Liquid Chromatography (HPLC) Analysis of Test Substance .....	5
HPLC/UV ANALYSIS .....	5
HPLC Method .....	6
Corrosion Characteristics .....	6
Preparation of Samples .....	6
Method(s) for Control of Bias .....	7
PROPOSED STATISTICAL METHOD(S) .....	7
RECORDS TO BE MAINTAINED .....	8
GLP Compliance .....	8
REPORT .....	8
Amendments and Deviations to the Protocol .....	9
PROTOCOL ACCEPTANCE .....	10



## **INTRODUCTION**

K32 is a fertilizer.

## **PURPOSE**

The purpose of this study is to determine the storage stability and corrosion characteristics of K32. The study will be conducted to meet the data requirements of guideline OPPTS 830.6317 (Storage Stability) and OPPTS 830.6320 (Corrosion Characteristics).

## **EXPERIMENTAL INFORMATION**

### ***SCHEDULE OF EVENTS***

Proposed Experimental Starting Date: September 2016  
Proposed Experimental Termination Date: September 2017

The actual starting and termination dates will be documented in the final report.

### ***SPONSOR***

Koch Agronomic Services, LLC  
2883 Miller Road  
Decatur GA 30035

### ***SPONSOR REPRESENTATIVE***

Eric Searcy  
Product Regulatory Manager  
Koch Agronomic Services, LLC  
2883 Miller Road  
Decatur GA 30035  
Phone: 770-593-6813  
Email: eric.searcy@kochind.com

### ***TESTING FACILITY***

Ricerca Biosciences, LLC  
AgChem Product Development  
7528 Auburn Road  
Concord, OH 44077

### ***STUDY DIRECTOR***

Penny Miner  
AgChem Product Development  
Ricerca Biosciences, LLC  
7528 Auburn Road  
Concord, OH 44077  
Phone: (440) 357-3718  
Fax: (440) 357-3654  
Email: penny.miner@ricerca.com





## **MATERIALS AND METHODS**

### ***STORAGE, DISTRIBUTION, AND RETENTION***

The test substance will be supplied by the Sponsor or their representative and will be stored at conditions specified by the Sponsor. The original container will be retained until the final report is issued and then discarded. Ricerca Biosciences will dispose of the unused test substance at the completion of the work, unless directed otherwise in writing from the Sponsor.

### ***TEST SUBSTANCE***

- **K32**

Composition:	Reaction products of NBPT with urea and formaldehyde
Batch/Lot Number:	55700-30-13
Analyzed Concentration:	Reaction product mixtures 80.3 wt%, NBPT 17.3 wt%, water 2.4 wt%
Manufactured by:	Ricerca Biosciences
Date of manufacture:	July 20, 2016
Appearance:	Off-white to pale yellow gel
Storage:	Refrigerated

### ***CHARACTERIZATION OF THE TEST SUBSTANCE***

It is the responsibility of the Sponsor to provide characterization of the test substance used in this study. The Sponsor will assume the responsibility of retention of a sample of the test substance as specified in 40 CFR 160.195.

## **JUSTIFICATION FOR SELECTION OF TEST SYSTEM**

The test procedures have been selected to comply with United States Environmental Protection Agency Product Properties Test Guidelines, August 1996. The test system is the test substance itself.

## **EXPERIMENTAL DESIGN**

Summary of tests to be performed are as follows:

Stability over a 1 year period will be determined by HPLC analysis. All HPLC data will be collected by Perkin Elmer Totalchrom™ (validated system). Microsoft Excel™ (non-validated software) may be used as to generate necessary statistics.

### ***OVERVIEW***

The following detail is provided as a guideline for the conduct of the study. Good scientific judgment may be applied to optimize the experimental results. The actual procedure will be recorded in the data and summarized in the final report.

All standard operating procedures pertaining to this study shall be available for inspection at Ricerca.



### ***STABILITY AT AN ELEVATED TEMPERATURE***

#### **Equipment**

- Constant temperature chamber
- General laboratory equipment: beakers, flasks, pipets, vials, glassware, etc.
- Liquid chromatographic system equipped with an autosampler, a UV/VIS detector, and a data acquisition system.

#### **Reagents**

- Acetonitrile, Methanol or organic solvent specified by the sponsor, HPLC grade or better
- Water, HPLC grade or better

### ***STABILITY STORAGE OF SAMPLE AT A TEMPERATURE OF $20 \pm 2$ °C***

#### ***PROCEDURE***

#### **Preparation of Samples**

The as-received test substance will be repackaged by equal distribution into replicate packages of the same construction and material as the commercial package (HDPE Bottle). The packages of test material will be placed in storage at 20 °C (+/- 2 °C) and 50% (+/- 5%) humidity. All packages of test material will be weighed prior to being placed into storage and upon being removed from storage. The temperature will be routinely monitored throughout the 1 year study.

#### **Physical Evaluation of Test Substance and Containers**

At each time interval (0, 3, 6, 9, and 12 months), the test substance will be examined for physical changes in appearance, e.g., color and clarity. The container itself will be examined for any evidence of physical degradation, e.g., cracking, pitting, or brittleness.

#### **High Performance Liquid Chromatography (HPLC) Analysis of Test Substance**

At the beginning of this study, duplicate aliquots of test material will be assayed. The assays will follow the procedures provided by the Sponsor. After 3, 6, 9, and 12 months of storage, samples will be gently hand shaken to assure homogeneity, and assayed in duplicate for active ingredient.

An aliquot will be withdrawn from each container for quantitation of active ingredient by HPLC. If any pair of duplicate determinations are in poor agreement, additional aliquots will be withdrawn and quantitated for active ingredient.

### **HPLC/UV ANALYSIS**

The stability of K32 heat treated test substances will be compared to the control sample stored at ambient temperature (~25 °C) to determine if any decomposition has occurred by the following HPLC/UV method. Samples should be prepared with 0.2 grams of sample in 1 mL of ACN/water 3:7.



**Storage Stability and Corrosion Characteristics of K32**  
**Document No.: 035237-0**

**HPLC Method**

Column: Restek Ultra C18, 3  $\mu$ m, 150 mm x 4.6 mm  
Mobile Phase A: Water  
Mobile Phase B: Acetonitrile  
Flow rate: 1 mL/min  
Injection volume: 15  $\mu$ L  
Detection: UV, 214 nm

**Gradient Method**

Step	Time	MP A	MP B	Curve
0	0.5	87	13	0
1	8.0	87	13	0
2	22.0	30	70	1
3	1.0	87	13	0
4	12.0	87	13	0

**CORROSION CHARACTERISTICS**

**Preparation of Samples**

Aluminum, zinc, copper, stainless steel, mild steel and brass coupons (1/2" x 3" x 1/16") will be washed with detergent, water, alcohol and dried in an oven thermostatically controlled to the specified temperature ( $50 \pm 2$  °C) and subsequently cleaned with absorbent paper (tissue paper).

After drying, aluminum, zinc, copper, stainless steel, mild steel and brass coupons will be weighed [a, (g or mg)] and placed in separate six small containers of the same composition as the commercial container (HDPE Bottle) with test item and one commercial container with test item will be used as control. These seven containers will be considered as one set. Similarly, five more sets will be prepared and will be stored at ambient storage condition for a period of 12 month and the maximum and minimum temperature along with the relative humidity (% RH) will be recorded. The container will be weighed initially at the time of storage and reweighed at each of the test intervals, prior to and after sampling. The samples will be drawn at various intervals [before storage (0 day) and at intervals of 3, 6, 9 and 12 months] and following test will be performed:

**(A) Corrosion Characteristics:**

1. Corrosion characteristics (perforation, darkening and leaking at the seam)
2. Appearance of the container (by visual: shape, color, cracks if any)
3. Corrosiveness

**Corrosiveness**

One set will be taken out at each interval and the following procedure will be followed:

The aluminum, zinc, copper, stainless steel, mild steel and brass coupons will be washed with detergent, water, alcohol and dried in an oven and subsequently cleaned with absorbent paper



**Storage Stability and Corrosion Characteristics of K32**  
**Document No.: 035237-0**

---

(tissue paper). After drying of the aluminum, zinc, copper, stainless steel, mild steel and brass coupons, visual changes on the metals (if any) will be recorded. The metals will be weighed [b, (g or mg)] again and the respective differences in their weights will be recorded for determination of corrosion rate [in mils per year (mpy)].

**4.4.2 Calculation of Corrosion Rate**

Corrosion rate will be calculated using the following formula:

$$\text{Corrosion Rate (mpy)} = \frac{K \times W}{A \times T \times D}$$

Where,

$$\begin{array}{ll} K = \text{Constant } (3.45 \times 10^6) & T = \text{Time of exposure (h)} \\ W = \text{Loss of weight (g)} & D = \text{Density (g/cm}^3\text{)} \\ A = \text{Area of metal (cm}^2\text{)} \end{array}$$

***METHOD(S) FOR CONTROL OF BIAS***

Bias will be controlled in the experiment by use of duplicate samples and by averaging multiple measurements.

**PROPOSED STATISTICAL METHOD(S)**

Appropriate statistical methods for the analysis and evaluation of the experimental data will be used at the discretion of the Study Director. Common statistical methods used to evaluate the precision and accuracy of the measurements may include (as appropriate): average, mean, coefficient of variation, standard deviation, relative standard deviation, and confidence interval. Outlier measurements can be evaluated per standard statistical procedures such as the "t test" or "Q test."

To improve data presentation and interpretation, and facilitate report preparation, the Study Director may apply computer programs for spreadsheets (e.g., Excel), graphics presentations (e.g., PowerPoint), and general standard statistics software.



## **RECORDS TO BE MAINTAINED**

Analysts shall document all experimentation such that an experienced scientist can reconstruct the work. Documentation shall include sample identifications, weighings, dilutions, calculations, etc. Additional documentation shall include instrumentation and equipment utilized during the study, as well as documentation of prepared reagents and solutions.

All study data shall be reviewed or verified and maintained in folders in the study activity file. Other comments, descriptions, calculations, correspondence, etc., shall be placed in the study activity file.

Upon conclusion of the study, copies of representative raw data (as appropriate), shall be submitted to the Sponsor. An accurate study file, including original raw data, shall be submitted to the Ricerca Biosciences, LLC Archives, 7528 Auburn Road, Concord, Ohio.

### ***GLP COMPLIANCE***

The described study will be conducted in accordance with the U.S. Environmental Protection Agency's "Good Laboratory Practice Standards" as published in 40 CFR Part 160. This study will be routinely examined by Ricerca Biosciences Quality Assurance Unit personnel for compliance of GLP, protocol, and SOPs.

## **REPORT**

A final report will be prepared at the conclusion of the study. The report shall include, but not necessarily be limited to, the following:

- Name and address of the facility performing the study and the dates on which the study was initiated and completed, terminated, or discontinued
- The approved protocol and any amendments to the original protocol
- Reference(s) to, and/or a detailed description of, all methods used
- Representative data generated while conducting the study, and representative transformations, calculations or operations performed on the data
- Identification of the test substances used in the study
- All deviations and changes from the protocol
- A description of all circumstances that may have affected the quality or integrity of the data
- Name and signature of the Study Director, the names of other scientists or professionals, and the names of supervisory personnel involved in the study
- Statistical methods employed for analyzing the data. A description of the transformations, calculations, or operations performed on the data, a summary and analysis of the data, and a statement of the conclusions drawn from the analysis
- Locations where raw data and the final report are to be stored
- The signed and dated statement by the Ricerca Quality Assurance Unit specifying the dates of study inspections and dates the findings were reported to the Study Director and Management, when applicable

Contains Proprietary Information: This document contains and makes reference to information that Koch Agronomic Services, LLC (KAS) considers to be commercially valuable. The fact that this document might be provided to a government agency by KAS, or made available to the public by a government agency, should not be construed to convey any right to any third-party to cite, rely upon, or otherwise make use of this document, or the data referenced herein, for any purposes.

Any use other than use by the United States Environmental Protection Agency is prohibited without the express written permissions of an authorized representative of KAS.



**Storage Stability and Corrosion Characteristics of K32**  
**Document No.: 035237-0**

---

- The signed and dated statement by the Study Director describing compliance with the Good Laboratory Practice Standards as specified in 40 CFR 160

***AMENDMENTS AND DEVIATIONS TO THE PROTOCOL***

All agreed upon amendments will be expressed in writing, and signed and dated by the Sponsor and the Study Director. Copies of the signed amendments will be returned to the Study Director and appended to the protocol.

The Study Director will communicate the nature of any deviations to the Sponsor. Deviations from the protocol, if any, will be documented and described in the final report.

Contains Proprietary Information: This document contains and makes reference to information that Koch Agronomic Services, LLC (KAS) considers to be commercially valuable. The fact that this document might be provided to a government agency by KAS, or made available to the public by a government agency, should not be construed to convey any right to any third-party to cite, rely upon, or otherwise make use of this document, or the data referenced herein, for any purposes. Any use other than use by the United States Environmental Protection Agency is prohibited without the express written permissions of an authorized representative of KAS.




**Storage Stability and Corrosion Characteristics of K32**  
**Document No.: 035237-0**

**PROTOCOL ACCEPTANCE**

**Study Title:** Determination of Storage Stability and Corrosion Characteristics of K32


**Document Number:** 035237-0

**Testing Facility:** Ricerca Biosciences, LLC  
7528 Auburn Road  
Concord, OH 44077

  
Penny Mijer  
Ricerca Biosciences, LLC

  
Date

  
Phillip Cassidy, Management  
Ricerca Biosciences, LLC

  
Date

  
Eric Searcy, Sponsor Representative  
Koch Agronomic Services, LLC

  
Date

Contains Proprietary Information: This document contains and makes reference to information that Koch Agronomic Services, LLC (KAS) considers to be commercially valuable. The fact that this document might be provided to a government agency by KAS, or made available to the public by a government agency, should not be construed to convey any right to any third-party to cite, rely upon, or otherwise make use of this document, or the data referenced herein, for any purposes. Any use other than use by the United States Environmental Protection Agency is prohibited without the express written permissions of an authorized representative of KAS.



## PROTOCOL AMENDMENT ONE

### Study Title:

Determination of Storage Stability and Corrosion Characteristics of K32

**Concord Biosciences Study Number:** 035237  
**Concord Biosciences Document Number:** 035237-0-1

**Testing Facility:**  
AgChem Product Development  
Concord Biosciences, LLC  
10845 Wellness Way  
Concord, Ohio 44077, USA

**Study Sponsor:**  
Koch Agronomic Services, LLC  
2883 Miller Road  
Decatur GA 30035



Contains Proprietary Information: This document contains and makes reference to information that Koch Agronomic Services, LLC (KAS) considers to be commercially valuable. The fact that this document might be provided to a government agency by KAS, or made available to the public by a government agency, should not be construed to convey any right to any third-party to cite, rely upon, or otherwise make use of this document, or the data referenced herein, for any purposes. Any use other than use by the United States Environmental Protection Agency is prohibited without the express written permissions of an authorized representative of KAS.



Protocol Amendment One / Storage Stability and  
Corrosion Characteristics of K32  
Document Number: 035237-0-1

---

## **ORIGINAL PROTOCOL SECTION**

**ALL PAGES WHERE THE NAME AND/OR ADDRESS APPEARS:**

Ricerca Biosciences, LLC  
AgChem Product Development  
7528 Auburn Road  
Concord OH 44077

## **CHANGE TO**

**Concord Biosciences, LLC**  
AgChem Product Development  
**10845 Wellness Way**  
Concord, OH 44077

## **REASON FOR CHANGE**

The name and address of the testing facility have changed.

## **EFFECTIVE DATE**

The effective date is the date that the amendment is signed by the Study Director.

## **ORIGINAL PROTOCOL SECTION**

**ALL INCIDENCES WHERE RICERCA.COM APPEARS IN AN EMAIL ADDRESS OF AN EMPLOYEE  
NOW WITH CONCORD BIOSCIENCES, LLC:**

penny.miner@ricerca.com

## **CHANGE TO**

penny.miner@concordbio.com

## **REASON FOR CHANGE**

The email addresses of the testing facility have changed.

## **EFFECTIVE DATE**

The effective date is the date that the amendment is signed by the Study Director.


Contains Proprietary Information: This document contains and makes reference to information that Koch Agronomic Services, LLC (KAS) considers to be commercially valuable. The fact that this document might be provided to a government agency by KAS, or made available to the public by a government agency, should not be construed to convey any right to any third-party to cite, rely upon, or otherwise make use of this document, or the data referenced herein, for any purposes. Any use other than use by the United States Environmental Protection Agency is prohibited without the express written permissions of an authorized representative of KAS.



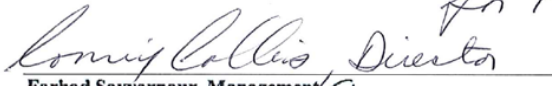
Protocol Amendment One / Storage Stability and  
Corrosion Characteristics of K32  
Document Number: 035237-0-1

## PROTOCOL AMENDMENT ONE ACCEPTANCE

**Study Title:** Determination of Storage Stability and Corrosion  
Characteristics of K32  
**Document Number:** 035237-0-1  
**Testing Facility:** AgChem Product Development  
Concord Biosciences, LLC  
10845 Wellness Way  
Concord, OH 44077

  
Penny Miner, Study Director  
Concord Biosciences, LLC

  
Date

  
Farhad Sayyarpour, Management  
Concord Biosciences, LLC

  
29-Sept-2017  
Date

  
Eric Searcy, Sponsor Representative  
Koch Agronomic Services, LLC

  
Date